

SPECIFICATION

(11) 294 253

NO DRAWINGS

(21) Application No. 27120/71 (22) Filed 19 April 1971

(31) Convention Application No. 103 805 (32) Filed 4 Jan. 1971 in

(33) United States of America (US)

(45) Complete Specification published 25 Oct. 1972

(51) International Classification C11D 3/08

(52) Index at acceptance C5D 6A2 6A5E 6A9 C1A D41 G12 G12D41 G4 G48



(54) USE OF SYNTHETIC CLAY CONTAINING NO LITHIUM AS SOIL ANTI-REDEPOSITION AGENT, IN DETERGENTS

(71) We, PFIZER INC., a Corporation organized under the laws of the State of Delaware, United States of America, of 235 East 42nd Street, New York 17, State of New York, United States of America, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

Modern day synthetic detergents are usually very effective in loosening soil particles in dirty clothing fabrics and removing the soil into the wash water during laundering operations, the soil being suspended in the aqueous detergent solution. However, some of this soil will be redeposited on the fabric during the laundering, causing a loss of whiteness in the fabric and a grey appearance.

One of the more common soil anti-redeposition (SARD) agents used commercially today in detergent formulations is carboxymethylcellulose (CMC) or its alkali metal salt, usually being sodium. Another common SARD agent used in commercial detergent formulations is polyvinylpyrrolidone (PVP). However, both SARD agents are relatively expensive, and in addition, do not seem to be uniformly effective against all types of fabrics; CMC not being uniformly effective on fabrics having synthetic fibers, and PVP not being totally effective on cotton fabrics.

In contrast to the prior art, it has been surprisingly found that SARD properties can be imparted to a detergent formulation, whereby the redeposition of soil upon a fabric from the aqueous washing solution of said formulation is substantially reduced, by incorporating in the formulation an effective amount of a compound having the formula

X(-) [Si₈Mg_{6-x}H_x(OH)₄O₂₀]. Na_x⁽⁺⁾

wherein X is from about 0.5 to 0.9. Prefer-

ably the compound is used in the formulation in a form wherein about 90% of the particles of the compound have a particle size smaller than about 325 mesh. More preferably, X is 0.7 in the general formula above and this compound is particularly effective on cotton fabrics and synthetic fabrics.

Most clay minerals, as found naturally, are in an impure state and the complete purification of some is difficult and expensive and, in some cases, impossible. Further, there are occasions on which the supply of a clay mineral of a particular chemical composition, either pure or impure, is insufficient. Thus, it is desirable to be able to manufacture synthetic clay-like minerals in a substantially pure form and of pure white color.

It is of particular interest to be able to manufacture synthetic clay-like minerals having rheological properties similar to or better than those of hectorite, as natural hectorite has valuable properties but large quantities of hectorite are not available. In any event natural hectorite is mixed with impurities the removal of some at least of which is extremely difficult. The naturally-occurring clay, hectorite, has the formula

$[Si_{8}Mg_{3,34}Li_{0.66}(OH)_{4}O_{20}]$. Na_{6,66}

wherein F may replace some OH. The clay-like materials used in the present invention have structural formulas very similar to natural hectorite, X in the general formula being 0.66, and H replacing Li in the formula for hectorite.

Two methods are known for synthesizing hectorite-type clay minerals. One is described in Granquist and Pollack, "Clays and Clay Minerals", National Academy of Science, National Research Council Publication, vol. 8, pp. 150—69 (1960). The other is described by Strese and Hofmann in Z. Anorg. Chem., 247, pp. 65—95 (1941).

The clay-like materials of the present inven-

10

15

25

geously prepared by means tion may be adva of a process which comprises:

forming an aqueous slurry from i. a water-soluble magnesium salt,

ii. sodium silicate, iii. sodium carbonate or sodium hydroxide and the aqueous slurry being formed by co-precipitation by slowly combining the said magnesium salt and the said sodium silicate and the said sodium carbonate or sodium hydroxide, with heating and agitation (e.g. 200°F.), the slurry containing steichiometric amounts of all the cations and anions that are desired to be present in the final product;

(b) taking the aqueous slurry so formed (e.g. after about 2-4 hours), washing it free from soluble salts, filtering, and adding aquecus NaOH to maintain an alkaline

solution; (c) Autoclaving the slurry (e.g. at a pressure about 100—200 psig and a corresponding steam temperature about 338-388°F.) for about 10-20 hours to crystallize the

synthetic mineral-like clay; and (d) drying and grinding the finished product. Alternatively, step (a) can be performed in a pebble mill without heating in which the slurry is formed by blending for about 1 hour and then processed according to steps (b), (c), and (d) above.

Many variations of the above process can be made by anyone skilled in the art.

Any suitable magnesium salt may be used for introducing the Mg cation into the solution such as magnesium chloride, magnesium sulfate, and magnesium nitrate. It is preferred to use MgSO₄. 7H₂O. The silicon cation may be introduced in the form of any suitable silicate. The sodium cation is introduced preferably in the form of Na₂CO₃, NaOH, or

The process for preparing the clay-like minerals of the invention has broad applicability in the preparation of compounds which are similar in structure to other naturally occurring clays (e.g. other montmorillonites). Thus by introducing the correct stoichiometric amounts of cations and anions in the initial slurry, it may be possible to prepare compounds having the general formula

$$\begin{array}{c} X(-)_x \\ M \\ [Si_sMg_{s-x}Li_xF_y(OH)4 - {}_yO_{2s}] \cdot \frac{M}{n} \end{array}$$

where M can be any cation having a valence, n, (e.g. K, Cu, Ca, Co, Al, Li, etc.).

The synthetic clay-like materials having the general formula

$$[Si_sMg_{6-x}H_x(OH)_4O_{2v}].Na_x^{(+)}$$

where X is from about 0.5 to 0.9 are preferred

for use as soil anti-rede on (SARD) agent in detergent formulations. Most preferably, not less than about 90% of the SARD agent particles in the detergent formulation have a particle size smaller than 325 mesh. Furthermore, it has been surprisingly found that when X is 0.7, this particular compound is more effective on cotton and synthetics than the commercial SARD agent, CMC, especially at the small particle size distribution mentioned above. The fine particle size is especially preferable for cotton in view of the normally short washing times (e.g. 10 minutes), to promote quick dispersability of the compound in the detergent wash solution for maximum effectiveness.

Of course, it is contemplated within the scope of the present invention that the claylike materials disclosed herein may be used in combination with commercial SARD agents such as CMC and PVP to improve their SARD properties.

The following typical wash test procedure is used to evaluate the effectiveness of the synthetic clay-materials as SARD agents in detergent formulations:

1. Turn the tergotometer heater switch on and adjust the thermostat to 120°F. Set agitator speed at 100 cycles per minute.

2. Weigh out one 1.50 gram of each detergent formulation sample (which may contain a SARD agent) to be tested.

3. Add each weighed amount to a tergotometer bucket filled with 975 ml. of tap water. Agitate this mixture 30 seconds.

4. Add 25 ml. of 2% Aquadag (registered Trade Mark) solution (made by mixing into 1000 ml. of deionized water 20 g. of Aquadag stock solution, which contains 22% solids of colloidal graphite in water) to each bucket containing detergent and water. Agitate this for 30 seconds.

5-A. Add to each bucket the following standard test fabric swatches identified with laundry ink.

Eight white pieces of 80×80 cotton (3" × 6") folded in thirds.

Four white pieces of Dacron/cotton 7406 WRL (3" × 6") folded in thirds.

5-B. Or add to each bucket the following 3" × 6" fabric swatches folded in thirds.

Dacron/cotton 7406 WRL Dacron/cotton 7406 Cotton 400W Dacron spun 754 AW Nylon spun 358 Dacron/cotton 7402A Acetate Jersey S/113 Spun Viscose S/266 Orlon—75 S/862	-2 pieces -1 piece -3 pieces -1 piece	
		

Total 12 pie ("Dacron" and "Orlon" are Registered 12 pieces 120 Trade Marks

ጸበ

100

6. Allow tergotome agitate 10 minutes. 7. After completion or cycles remove fabrics

and agitators. Squeeze excess liquid from swatches.

3

8. Add 1000 ml. of 100°F, water to each bucket. Place the swatches in same tergorometer bucket and rinse for 5 minutes at 100 cycles per minute agitation.

9. Remove swatches from the bucket and 10 squeeze the excess water out and dry.

10. After the fabric is completely dry and conditioned to room temperature reflectance readings are taken using a standard Reflectometer. Using white tile as the reference standard, take the reading through a single thickness of 80 × 80 cotton. To take readings on Dacron/cotton arrange the four swatches from a bucket in sandwich form for reflectance measurements. When comparing detergent formulations, lower reflectance readings after washing indicate lower SARD effectiveness, on a given test fabric.

In the above description of a typical wash test procedure, it should be noted that a tergotometer is a standard instrument for testing detergency efficiency, usually consisting of four numbered 1.5 litre pots containing agitators which are used to simulate typical laundering conditions.

Also, in step 5A or 5B the standard test fabrics used are identified by a number assigned by the supplier of these fabrics-Testfabrics, Inc., New York City-and are as follows:

1. Dacron/Cotton—style 7406 WRL— 65/ 35 shirting with permanent press finish.

2. Cotton-style 400 W-Bleached 80 x 80 cotton print cloth.

3. Dacron/Cotton — Styde 7406 — 63/35 shirting.

4. Acetate Jersey — Style 113 — All delustered filament.

5. Spun viscose—Style 266—Challis (print cloth).

6. Dacron type 54-Style 754 AW-100% spun fabric.

7. Orlon-75-Style 862-Sand Weave.

 8. Nylon spun 6.6—Style 358.
 9. Dacron/cotton — Style 7402A — 65/35 poplin (raincoat weight).

10. Dynel — Style 902 — modified acrylic (this fabric sample was not used in all the tests).

Typically, the clay materials of the invention have relatively high surface areas compared to natural clays, being about 100-600 meters²/

It has been known in the past that natural clays (e.g. Ben A Gel and sodium bentonite BH—200) have soil suspending properties, however as shown in Example VIII, their SARD properties are truly inferior to the clay materials of this invention. ("Ben A Gel" is a Registered Trade Mark).

65 Effective amounts of SARD agents in com-

mercial detergent formulation typically 0.5—1.5%, based on the weight of the formulation. Preferably, about 1% by weight of the compounds of the invention are incorporated as SARD agent.

Use of the compounds of the invention in detergent formulations contributes to the antistatic properties of the fabric.

The following examples are provided for illustrative purposes and should not be interpreted as limiting the invention, the scope of which is defined by the appended claims.

Example I A) A compound (where X is 0.7) having the formula

 $[Si_8Mg_{5.3}H_{0.7}(OH)_4O_{20}]$. Na_{0.7}

is prepared by first forming a slurry of the ingredients by co-precipitation as follows:

A stirred solution containing 5310 grams of MgSO.7H₂O dissolved in 25 liters of water and heated to about 205°F. in a 60 liter tank by means of steam coils, is precipitated over a 29 minute period with an alkaline solution having a temperature of about 140°F. and prepared by dissolving 6810 grams of N sodium silicate (8.6% Na₂O, 28.6% SiO₂) and 1285 grams of Na₂CO₂ in 25 liters of hot water. At the end of the precipitation period the precipitate slurry temperature is about 198°F. This is rapidly increased to and maintained at about 206°F. while the slurry is digested for a period of 1-1/2 hours during which time additional gas, taken to be CO, is evolved from the slurry. The slurry is then filtered and the resulting filter cake washed with 30 liters of water and allowed to drain on the filter. The washed filter cake, having a total weight of about 22,390 grams, is stirred to a viscous fluid into which is stirred a solution containing 350 grams of NaOH in 750 ml. of water. Then this material is placed in pans and subjected to elevated temperature, about 365°F., at the corresponding gauge pressure, about 150 psi, in a steam operated, horizontal autoclave for about 16 hours. The autoclave product cake is cut into small pieces, dried, disintegrated in a hammer mill and then pebble mill ground to a fine powder (to about 5% plus 325 mesh).

B) Alternatively, the compound having the 115 same above chemical formula is prepared by first forming the slurry by pebble milling as follows;

Seven batches are made as follows and blended. 200 ml. of water are stirred into 478 grams of MgSO4 . 7H2O crystals. To this is added a solution containing 612 grams of N sodium silicate, 91 grams of NaOH and 300 ml. of water. The batch is stirred to a fluid, gritty mass. The blend is then ground in a vertical, vibrating pebble mill for a period of one hour. The mill is discharged and rinsed

30

35

20

with 6300 ml. of ter. The resulting, stirred slurry is filtered and the filter cake is washed with 14,000 ml. of water and allowed to drain on the filter. The washed filter cake, having a total weight of about 7080 grams is stirred to a viscous fluid into which is stirred a solution containing 123 grams of NaOH in 350 ml. of water. This material is autoclaved as above and dried. Two batches of dried product are produced, blended and ground to a fine powder as above.

Example II

Th procedure of Example IA or IB is followed to produce a compound having the same chemical formula but the final product is ground to smaller than 60 mesh and is not as fine as 325 mesh.

Example III A compound having the formula

 $[Si_{s}Mg_{s,1}H_{a,9}(OH)_{4}O_{20}] \cdot NA_{a,9}$

is prepared by follow the procedure of Example IB, except that:

460 grams of MgSO₄. 7H₂O are used instead of 478 grams; 83.3 grams of NaOH are used instead of 91 grams; and the final product is ground to smaller than 60 mesh rather than 325 mesh.

Example IV A compound having the formula

 $[Si_8Mg_{3.5}H_{0.5}(OH)_4O_{20}]$. $Na_{9.5}$

is prepared by following the procedure of Example IB, except that:

495 grams of MgSO₄. 7H₂O are used instead of 478 grams; 93.8 grams of NaOH are used instead of 91 grams; and the final product is ground to smaller than 60 mesh rather than 325 mesh.

Example V
The standard wash procedure previously described is followed in which the following detergent formulation samples in Table 1 are tested for SARD properties:

TABLE 1

	Sample No.						
	1	2	3	- 4	5	6	
Colgate Detergent Base	1.485 g.	1.485 g.		1.485 g.	1.485 g.	1.485 g.	
C.M.C.		0.015 g.					
Ajax (Registered Trade A Detergent	Aark)		1.5 g.				
P.V.P. K-30				0.015 g.			
Compound prepared in Example IA					0.015 g.		
Compound prepared in Example IB					·	0.015 g.	

Note in Table 1 that Ajax detergent has the following composition shown in Table 2.

	TABLE 2		com
45	Anionic surfactant	10 wt. %	ΑÎ
_	Nonionic surfactant	2	P
	Soap (e.g. sodium or potassium		
	stearate)	. 2	CI
	Sodium tripolyphosphate	35	an
50	Sodium Silicate	7	
	SARD agent (mixture of CMC as	nd	TI
	PVA) \	1	of th
	Sodium sulfate	3335	Table
	Water	8—10	to st
			in the

TARIE 2

The Colgate detergent base has the same composition as Ajax, but with no SARD agent.	5 5
Also, note that, PVP/K—30 means polyvinylpyrrolidone (Avg. mol. wt. 40,000); CMC means carboxymethylcellulose; and PVA means polyvinyl alcohol.	

The following reflectance readings for each of the six samples previously mentioned in Table 1 are given for fabric samples according to step 5A (Table 3) and step 5B (Table 4) 65 in the standard wash test procedure.

TABLE 3

	Average Reflectance	Reflectance after Washing					
Fabrics	Before Washing	1	2	3	4	5	6
Dacron/cotton — 7406 WRL 1st Piece	89.13	66.07	69.14	73.06	78.09	74.38	74.01
Dacron/Cotton — 7406 WRL 2nd Piece	88.92	65.94	68.06	73.19	79.21	74.79	74.21
Dacron/Cotton — 7406 WRL 3rd Piece	89.16	65.58	68.19	72.53	78.62	73.57	74.50
Dacron/Cotton — 7406 WRL 4th Piece	89.02	67.47	69.27	73.64	77.75	74.58	74.94
Cotton — 400 W 1st Piece	94.08	41.37	61.34	66.28	52.80	63.56	59.20
Cotton — 400 W 2nd Piece	94.39	43.60	60.01	64.02	52.14	74.56	60.60
Cotton — 400 W 3rd Piece	94.22	44.11	61.94	64.61	52.98	63.97	60.05
Cotton — 400 W 4th Piece	94.18	45.26	61.63	65.35	52.11	64.82	61.54
Cotton — 400 W 5th Piece	94.97	43.55	61.42	65.13	52.10	64.91	60.92
Cotton — 400 W 6th Piece	95.03	43.39	61.87	65.10	52.99	64.60	61.08
Cotton — 400 W 7th Piece	95.21	43.50	60.92	64.73	51.87	63.91	61.19
Cotton — 400 W 8th Piece	94.20	43.36	60.80	65.75	52.11	64.41	60.84

TABLE 4

	Average	Reflectance after Washing					
Fabrics	Reflectance Before Washing	1	2	3	4	5	6
Dacron/Cotton — 7406 WRL 1st Piece	91.69	69.19	70.51	76.22	78.09	77.21	77.94
Dacron/Cotton — 7406 WRL 2nd Piece	91.18	70.52	72.03	78.10	79.21	77.37	78.21
Dacron/Cotton — 7406	92.61	41.84	44.65	52.19	62.59	72.71	69.22
Cotton — 400=V	94.19	46.52	64.72	66.97	53.36	64.51	60.22
Cotton — 400=V 2nd Piece	94.30	47.08	65.45	64.49	53.53	65.77	59.92
Cotton — 400 W 3rd Piece	93.71	45.98	63.90	67.22	52.27	64.28	60.91
Dacron Spun 754 AW	90.86	77.67	77.95	81.31	82.03	82.18	83.34
Nylon Spun 358	89.19	73.02	75.21	77.39	82.84	78.28	82.29
Dacron/Cotton 7402A	87.43	27.99	27.05	28.28	31.07	56.41	51.81
Acetate Jersey S/113	90.11	31.34	32.25	48.32	73.65	73.37	68.80
Spun Viscose S/266	91.75	68.21	73.84	78.56	77.56	80.90	82.92
Orlon—75—S/862	89.89	83.32	81.77	83.25	84.32	84.83	84.14

Example VI

The standard wash procedure is similarly followed as in Example V, except that step 5B fabrics are only tested and the detergent formulation tested contains 1.485 g. of Colgate

detergent base and 0.015 g. of the compound as prepared in Example III. Reflectance values before and after washing are presented in Table 5 below.

TABLE 5

	Reflectance Before Washing		Reflectance after Washing	
	Dacron/Cotton 7406 WRL—1st piece	92.17	Dacron/Cotton 7406 WRL—1st piece	73.05
	Dacron/Cotton 7406 WRL—2nd piece	91.84	Dacron/Cotton 7406 WRL—2nd piece	74.96
	Dacron/Cotton 7406	92.42	Dacron/Cotton—7406	44.28
5	Cotton 400=W	95.16	Cotton—400 W	46.60
	Cotton 400=W	95.16	Cotton—400 W	43.44
	Cotton 400=W	95.16	Cotton—400 W	43.44
	Dacron 754=W	89.49	Dacron 754 W	62.14
	Nylon 358	89.99	Nylon 358	78.26
10	Dacron/Cotton 7402 A	90.37	Dacron/Cotton—7402 A	26.69
	Acetate Jersey S113	89.37	Acetate Jersey—S113	30.51
	Spun Viscose S266	89.80	Spun Viscose—S266	69.85
	Orlon75S862	87.19	Orlon—75—S—862	83.39

Example VII
The standard wash procedure is similarly followed as in Example V, except that step 5A fabrics are only tested and the detergent formulation tested contains 1.485 g. of Colgate 15

detergent base and 0.015 g. of the compound as prepared in Example IV. Reflectance values before and after washing are presented in Table 6 below.

TABLE 6

	Reflectance Before Washing	Reflectance After Washing
Dacron/Cotton — 7406 WRL 1 piece	92.11	75.33
Dacron/Cotton — 7406 WRL 2nd piece	91.83	77.41
Dacron/Cotton — 7406 WRL 3rd piece	92.16	76.46
Dacron/Cotton — 7406 WRL 4th piece	90.80	76.29
Cotton — 400 W — 1st piece	93.99	55.56
Cotton — 400 W — 2nd piece	93.64	56.35
Cotton — 400 W — 3rd piece	94.19	55.81
Cotton — 400 W — 4th piece	94.82	55.93
Cotton — 400 W — 5th piece	94.16	56.02
Cotton — 400 W — 6th piece	94.13	56.19
Cotton — 400 W — 7th piece	94.21	55.87
Cotton — 400 W — 8th piece	94.72	56.23

25

Example VIII
This example compares the SARD effectiveness in detergent formulations of natural clay materials such as Ben A Gel (see data in

Example IX) and Sodium Bentonite BH—200 (see data in Example IX) against the compound as prepared in Example II.

The standard wash procedure is similarly

followed as in Example V, except that step 5B fabrics are only tested and the detergent formulations tested contain 1.4888 g. of Colgate detergent base and 0.0112 (0.75%) of SARD agent each of 3 samples as follows:

Sample 1: SARD as is compound of Example II
Sample 2: SARD agent is Ben A Gel
Sample 3: SARD agent is Sodium Bentonite BH—200

Reflectance values before and after washing are presented in Table 7 below.

TABLE 7 Reflectance Before Washing

	1	2	3
Dacron/Cotton 7406 WRL-1	90.36	90.70	91.65
Dacron/Cotton 7406 WRL-2	90.87	90.91	91.85
Dacron/Cotton 7406	91.44	91.07	92.28
Cotton 400 W—1	92.97	93.90	95.54
Cotton 400 W-2	93.04	93.58	94.88
Dacron Spun 754 AW	89.24	89.27	89.53
Nylon Spun 358	90.58	90.73	90.28
Dacron/Cotton 7402 A	91.00	91.96	91.99
Acetate S/113	92.21	93.09	93.89
Viscose S/266	92.25	92.26	92.18
Orlon—75—S/862	86.60	88.15	87.19
Dynel (Registered Trade Mark) S/902	85.23	85.93	86.60

Reflectance After Washing

	1	2	3
Dacron/Cotton 7406 WRL-1	79.88	78.80	76.62
Dacron/Cotton 7406 WRL-2	80.93	76.29	76.75
Dacron/Cotton 7406	68.71	49.84	56.58
Cotton 400 W—1	55.39	45.39	48.32
Cotton 400 W—2	54.38	42.39	47.42
Dacron Spun 754 AW	82.74	64.87	71.71
Nylon Spun 358	79.78	74.28	73.35
Dacron/Cotton 7402A	43.04	26.05	27.96
Acetate S/113	48.94	22.24	26.43
Viscose S/266	82.65	67.50	73.13
Orlon—75—S/862	84.34	79.94	81.08
Dynel S/902	80.67	77.55	79.05

EXAMPLE IX

TABLE 8

X-Ray Diffraction and Nitrogen Surface Area Data

Sample	Crystalline Size Å	Surface Area m ² /g.
Compound from Example IA	56	109
Compound from Example IB	68	240
Compound from Example III	<50	_
Compound from Example II	_	537
*Ben A gel	200	52
*Sodium Bentonite, BH—200	238	31

^{*}Typical Analysis

Note that compounds prepared in Example I and Example II were prepared in a similar manner except that the autoclave product of Example II was washed prior to drying. It is therefore believed that the surface area listed for II is a true figure for the actual clay product and that the surface area for the clay portion of I should be much higher but was masked by the presence of the sodium

hydroxide in these samples which was not washed away.

Crystalline size is expressed as a dimension in terms of angstrom units of length. The crystalline is bounded by a change in electron density and the size is measured in a direction perpendicular to the characteristic 060 crystallographic plane of the (montmorillonite) unit cell.

Sodium Bentonite, BH-200 Ben A Gel SiO₂ 56.5 64.72 Al_2O_3 0.2 20.82 Fe₂O₃ 0.2 3.44 TiO₂ 0.14 MgO 25.8 2.38 CaO 2.8 0.49 Na₂O, K₂O 2.6 2.92 Li₂O 1.1 $\mathbf{C}\mathbf{I}$ 2.5 F 1.0 Ignition Loss 7.7 4.84 100.4 99.75

The standard wash procedure previously described is followed in which detergent formulation samples containing CMC, the compound prepared by Example IA, and the compound prepared by Example IB are evaluated for SARD effectiveness at varying

concentrations of the above SARD agents—no SARD, 0.25%, 0.50%, 0.75%, and 1.00% (all in weight % based on total weight of 1.5 grams for detergent formulation plus SARD agent). Table 9 shows the results based on reflectance readings before and after washing.

TABLE 9

Fabrics	Average Reflectance Before Washing	No S.A.R.D.	0.25% C.M.C.	0.25% Ex. ÍA	0.25% Ex. IB	0.50% C.M.C.
7406 WRL—1 Dacron-Cotton	91.08	68.28	66.30	69.91	70.17	66.10
7406 WRL—2 Dacron-Cotton	91.22	68.03	67.07	70.68	68.62	67.87
7406 WRL Average	91.15	68.15	66.69	70.29	69.39	66.99
7406 Dacron-Cotton	91.32	41.84	42.75	59.75	55.05	44.28
400 W—1 Cotton	93.90	50.81	54.81	54.28	52.48	60.25
400 W-2 Cotton	94.22	48.08	56.40	56.11	50.89	60.49
400 W—3 Cotton	93.89	49.30	55.19	55.81	49.87	60.09
400 W-4 Cotton	94.13	49.61	55.55	53.87	48.59	61.02
400 W Average	94.03	49.45	55.49	55.02	50.46	60.46
754 AW Spun Dacron	89.78	76.82	78.74	78.73	80.08	77.61
358 Spun Nylon	89.95	71.94	72.89	78.02	78.51	74.13
7402 A Dacron-Cotton	88.19	27.33	27.35	41.24	36.92	23.19
S/113 Acetate Jersey	91.11	30.43	31.23	63.75	57.70	30.88
S/266 Dynel	84.90	63.00	69.44	76.11	73.49	70.72

Reflectance After Washing

Fabrics	0.50% Ex. IA	0.50% Ex. IB	0.75% C.M.C.	0.75% Ex. IA	0.75% Ex. IB	1.00% C.M.C.	1.00% Ex. IA	1.00% Ex. IB
7406 WRL—1 Dacron-Cotton	71.16	71.02	67.95	72.91	72.92	68.49	72.78	73.95
7406 WRL—2 Dacron-Cotton	71.02	71.31	68.03	73.05	73.05	68.90	72.48	74.01
7406 WRL Average	71.09	71.16	67.99	72.98	72.99	68.70	72.63	73.68
7406 Dacron-Cotton	66.04	62.44	46.08	71.04	64.03	46.01	71.23	68.19
400 W—1 Cotton	59.59	54.43	64.91	68.15	57.17	61.26	68.48	62.40
400 W—2 Cotton	59.19	54.91	65.07	68.95	57.02	68.97	68.31	63.41
400 W—3 Cotton	59.73	54.87	64.83	68.34	56.90	67.55	69.20	62.91
400 W—4 Cotton	59.22	54.30	64.79	68.20	56.31	68.01	69.02	63.02
400 W Average	59.43	54.65	64.90	68.41	56.85	68.19	68.75	63.68
754 AW Spun Dacron	80.72	80.81	77.93	80.12	80.38	78.12	83.13	79.59
358 Spun Nylon	76.51	76.71	72.55	78.05	74.71	75.85	82.87	79.42
7402 A Dacron-Cotton	46.81	42.67	27.92	54.15	44.74	26.16	59.17	49.76
S/113 Acetate Jersey	73.33	67.72	37.82	76.56	66.29	37.40	79.90	71.83
S/266 Dynel	77.67	78.38	73.42	76.60	80.40	75.60	74.01	82.51

WHAT WE CLAIM IS:—
1. A method of washing fabrics with an aqueous detergent solution, whereby the redeposition of soil upon the fabric from the aqueous solution is substantially reduced, which comprises incorporating in said detergent a compound having the formula:

10 wherein

X is from 0.5 to 0.9

2. The method of claim 1 wherein about

90% by weight of the particles of said compound have a particle size smaller than 325 mesh (U.S. Sieve Standard).

3. The method of claim 2 wherein X is 0.7.

4. The method of claim 3 wherein said fabric comprises cotton.

5. The method of claim 3 wherein said fabric comprises a synthetic fabric.

6. The method of claim 3 wherein said fabric comprises a mixture of cotton and a synthetic fabric.

7. A method according to claim 1 substantially as described.

8. A detergent when used in the method of

15

claim 1 to which there has been imparted soil anti-redeposition properties substantially as described.

STEVENS, HEWILLT & PERKINS, Chartered Patent Agents, 5, Quality Court, Chancery Lane, London, W.C.2. Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1972.

Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

☐ BLACK BORDERS	
☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES	
☐ FADED TEXT OR DRAWING	٠
☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING	
☐ SKEWED/SLANTED IMAGES	
☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS	
GRAY SCALE DOCUMENTS	
☐ LINES OR MARKS ON ORIGINAL DOCUMENT	
☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY	•

IMAGES ARE BEST AVAILABLE COPY.

OTHER:

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.